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RPPR Final Report
as of 19-Nov-2018

Agency Code:

Proposal Number: 70029CHRIP **Agreement Number: W911NF-17-1-0250**
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STEM Degrees: **STEM Participants:**

Major Goals: Acquire, Install, and demonstrate operation of a Micromeritics Autopore V mercury porosimeter.

Accomplishments: See uploaded document.

Training Opportunities: During the installation of the porosimeter, students were trained on safe operation by engineers from Micromeritics.

Results Dissemination: Nothing to Report

Honors and Awards: Nothing to Report

Protocol Activity Status:

Technology Transfer: Nothing to Report

RPPR Final Report
as of 19-Nov-2018

Micromeritics AutoPore V report

PI: Matthew Green, Ph.D.

Co-PIs: Jerry Lin, Ph.D. and César Torres, Ph.D.

1. Installation

The instrument was installed on 3/21/2018 by a Micromeritics service engineer. Ventilation duct was installed as well to ensure the excess mercury vapor can be removed. Figures 1 to 3 show the present placement and ventilation of the instrument.

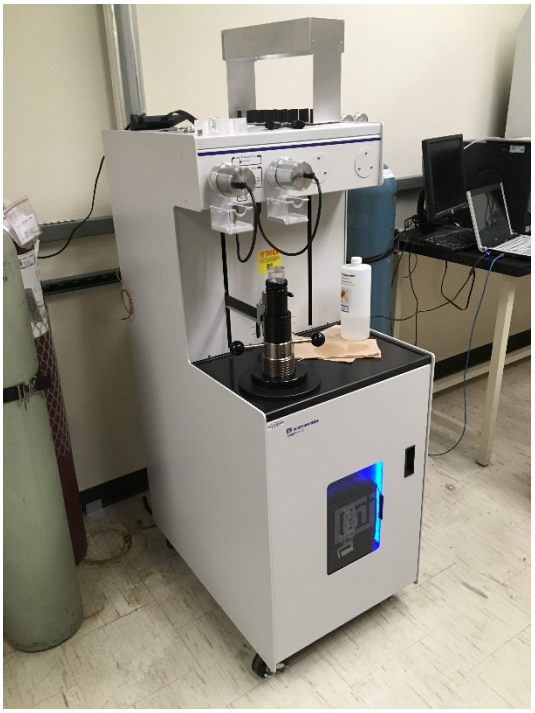

Figure 1. Micromeritics AutoPore V mercury porosimeter.

Figure 2. Ventilation duct installed by ASU facilities.

3. The ventilation connects the instrument to a venting hood.

2. Instrument operation

The instrument uses nitrogen to pressurize the mercury and measures the volume of mercury intrusion to obtain the information about the pores (pore volume, pore size distribution, porosity, etc.) of a porous material. To perform the measurement, powder or bulk solid sample must be placed in the penetrometer (sample tube), as shown in Figure 4. Penetrometers with different sizes and properties are included in the accessory kit. Depending on the sample or measurement conditions, a specific type of penetrometer may be selected. The handling of mercury is restricted in the venting hood to avoid mercury vapor release or mercury spill. A large metal tray is also used to avoid mercury spills.

Figure 4. Penetrometers for mercury porosimetry. Left: suitable for larger samples. Right: suitable for powders.

Figure 5. Sample preparation mercury handling area in a venting hood.

To this date a number of measurements have been conducted. The instrument has been working effectively and therefore accurate results were obtained. The software can generate reports and data graph to provide the users with instant preliminary data analysis without data processing, as shown in Figure 6. Some representative tests we have done are listed in Table 1:

Summary

Total intrusion volume at 61,000.00 psia:	0.1068 mL/g
Total pore area at 61,000.00 psia:	4.344 m²/g
Median pore diameter (volume) at 1,869.76 psia and 0.053 mL/g:	96.73 nm
Median pore diameter (area) at 2,051.08 psia and 2.172 m²/g:	88.18 nm
Average pore diameter (4V/A):	98.30 nm

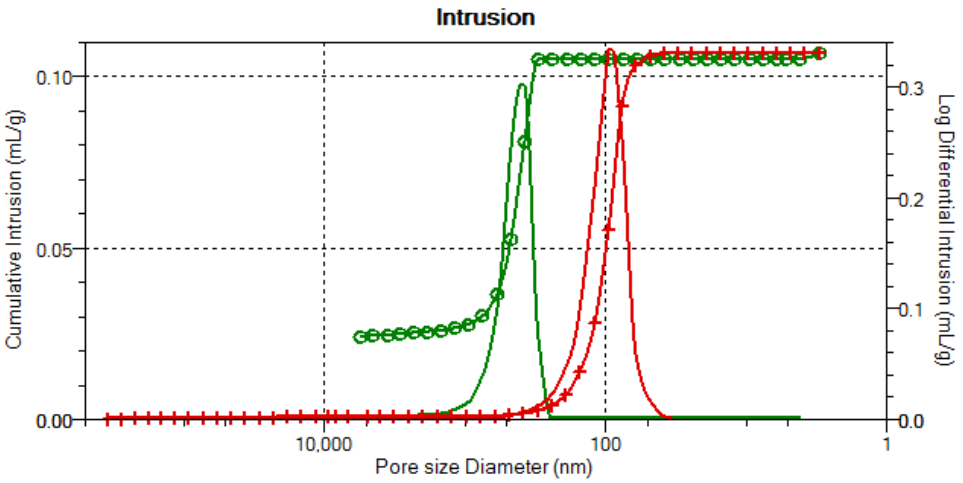

Figure 6. An example of report generated from the AutoPore V software (sample: YSZ).

Table 1. Examples of test results

Sample name	Original sample geometry	Total intrusion volume (ml/g)	Total pore area (m^2/g)	Average pore diameter (nm)	Porosity (%)
Y-Sm-Bi oxide – Sm-Ce-oxide dual-phase ceramic	Ceramic tube	0.0667	0.44	606.66	28.0
Y stabilized ZrO_2	Ceramic disc	0.1068	4.344	98.30	--
AC12 coated on Li-Ti-oxide	Ceramic thin film coated on electrode	0.4176	1.002	1667	--
Silica coated on Li-Ti-oxide	Ceramic thin film coated on electrode	0.4212	5.823	289.37	--
TiO_2-bohemite	Ceramic powder	0.1746	0.935	747.01	

As shown in Table 1, due to improper setting of parameter during the measurements, porosity data was not obtained. However, the reliability of all other data is not affected. It should be noted that if the sample can be wetted by mercury or the sample is compressible, it is not suitable to use this instrument for measurement. The following data is an example of measurements taken with a cellulose sample.

Summary Report

Intrusion Data Summary

Total intrusion volume at 60,217.89 psia:	0.6605 mL/g
Total pore area at 60,217.89 psia:	0.079 m^2/g
Median pore diameter (volume) at 6.08 psia and 0.330 mL/g:	29,768.44 nm
Median pore diameter (area) at 6.69 psia and 0.040 m^2/g:	27,038.62 nm
Average pore diameter (4V/A):	33,408.61 nm

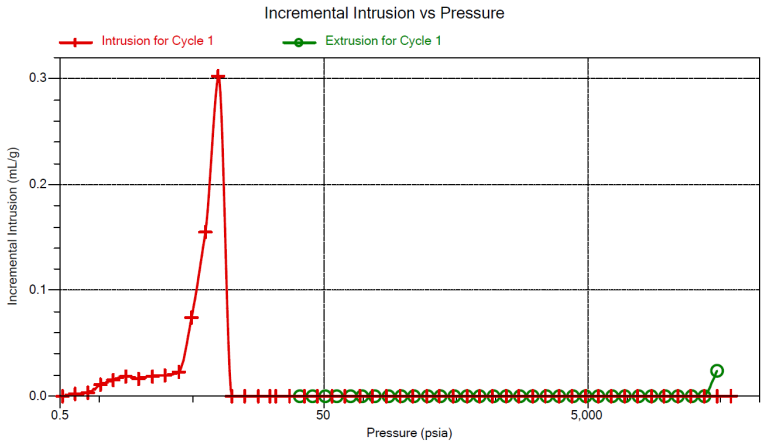

Figure 7. Measurement of cellulose sample.

Because of the compressibility of the sample, the mercury cannot intrude the pores when pressure is applied. Instead, the sample was compressed and deformed therefore the obtained data for pore size was too large and not reasonable.

Summary:

The equipment was successfully installed, including safety modification to the room, and is operating normally. This tool will be a great asset to characterize the porosity of samples prepared in support of DoD and ARO funded research programs.